MECHANISMS OF STRENGTHENING AND FRACTURE IN COMPOSITE MATERIALS

First Progress Report

To NASA

June 1, 1964 - November 30, 1964

Contract No. NSG-622

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INTRODUCTION

This report describes research activities that were performed during the initial phase of a program designed to investigate the mechanism of strengthening and fracture of composite materials. Three different types of composites are currently under investigation on separate but inter-related projects: 1) composites containing large volume fractions of a hard, brittle phase discontinuously distributed in a soft, ductile matrix; 2) composites containing an infinitely soft phase (in the form of drilled holes) dispersed in a matrix that can be either ductile or brittle, depending on test temperature and grain size; 3) lamellar composites containing soft and hard phases and pre-induced cracks in either the soft or the hard phase. The first project is primarily experimental, the second is both experimental and theoretical, and the third is entirely theoretical. These projects are described separately in the following sections.

1) <u>The Mechanism of Strengthening and Fracture in Composites Containing Hard Particles Dispersed in a Ductile Matrix</u> (with Darel Hodgson, graduate student).

It is believed that in systems such as these fracture occurs when inhomogeneous strains in the soft matrix can produce stress concentrations at the matrix-particle interfaces which are able to nucleate cracks in the hard particles[1]. At present, it is not known whether a critical tensile stress or strain concentration is required for crack nucleation. This project is designed to investigate this problem, for various volume fractions, sizes and shapes of hard particles dispersed in a ductile matrix.

The composite containing titanium carbide dispersed in a nickel-molybdenum matrix was originally chosen for investigation. During the summer of 1964, Mr. Hodgson worked at the Scientific Laboratory, Ford Motor Company. He worked about 25% of his time in Dr. Michael Humenick's group learning how to prepare this composite, which had originally been developed by Humenick and his co-workers. The principal advantage of this system, as compared to the conventional tungsten carbide-cobalt system used in previous investigations[1] of fracture in cermets is that good particle-matrix wetting is achieved so that homogeneous dispersions can be obtained. This does not seem to be the case in the tungsten carbide-cobalt system[2].

The composites, containing 50-75 volume percent titanium carbide, will be prepared by liquid phase sintering. All powders, milling materials, and additional equipment have been purchased on a grant from the Ford Foundation. These materials have been received and the powders are ready for milling and pressing.

The design of the die which will be used to press the dog-bone tensile specimens is presently under consideration. Mr. Hodgson discussed this problem with the Haller Company, Northville, Michigan, during the summer. It is anticipated that the die will be received within two months.

The specimens will be pre-sintered 650°C in a hydrogen gas train furnace which has been built and tested. Final sintering will be carried out under vacuum at 1370°C. The vacuum system and chamber for final sintering has been constructed and is ready for operation. Sintering will be done by induction heating using molybdenum heat shields. The final sintering susceptor and heat shields are half completed and should be ready to operate in about two months when the die arrives. Consequently, we expect to begin preparing samples for investigation about the first of February, 1965.

Meanwhile, Mr. Hodgson has been setting up the micro-strain equipment which will be used in the investigations of microcrack density as a function of stress and strain. A cryogenic device will be required, since measurements will be carried out at various low temperatures. This can be employed most easily by first constructing a "bridge", under the cross head of the Instron tensile testing machine, which holds one end of the specimen being strained in tension; the other end of the specimen is attached to a pull rod. The cryogenic device is then set around the bridge.

Both the bridge and the grips to hold the tensile specimen have been constructed. The cryogenic device will consist of a chamber containing dry helium gas surrounded by a dewar flask containing baths maintained at desired temperatures between 77 and 293°K. The micro strain measurements will be performed with a Tuckerman gauge and collimeter, both of which have been received. The gauge operates by the rotation, during strain, of a mirror which reflects an incoming beam of light at an angle that is a function of the amount of elongation of the specimen. Consequently, the chamber surrounding the specimen will contain a window through which light can be transmitted. Dry helium gas is used as a coolant to prevent the windows from fogging up during operation. This chamber is half completed at this time.

The initial measurements of microstrain in composite materials will be performed on hypo-eutectoid steels while any "bugs" are being worked out of the system. Steels containing 1% manganese and 0.2, 0.4 and 0.6% carbon will be tested. These alloys have been received and tensile specimens have been prepared from all three compositions. Various annealing treatments are currently being investigated to determine the best treatment for preparing composites containing uniform dispersions of fairly large cementite particles in the ferrite matrix. Microcrack density measurements will be made by obtaining replicas of the electo-polished specimen surface following each increment of microstrain. The replicas will be examined optically at magnifications of 1000 X and microcrack densities determined as a function of stress and/or microstrain. This non-destructive technique, developed by McMahon[3] during his investigation of fracture in ferrite, permits the determination of crack density as a function of stress and/or microstrain to be made on one specimen. It is more reliable than the older procedure of straining different samples different amounts, since all measurements are made on the same sample. Various replicating materials, such as parlodion, fax-film, wax and others, are currently being applied to the steel specimens to determine the best technique for use in the investigation.

Since the annealing treatments, cryogenic chamber, and proper replicating technique should be developed within one month, it is anticipated that initial measurements will begin on January 1, 1965. The next progress report will contain diagrams of the apparatus used in the investigation, as well as some results of the microstrain studies of the ferrite-cementite composite.

2) The Mechanism of Fracture in Composites Containing Small Holes Drilled in a Ductile or Brittle Matrix (with Charles Rau, graduate student).

Johnston, et al.,[4] have shown that cavities form at the interfaces between alumina particles and the silver chloride matrix during crack propagation in an $AgCl$-Al_2O_3 composite. These cavities relax tri-axial stresses at the tips of advancing cleavage cracks so that additional plastic deformation occurs during fracture and the energy absorbed in fracture increases. Consequently, the toughness of the composites is greater than that of the AgCl alone, and the notch impact transition temperature can be decreased by as much as 70°C when 2.5 volume per cent Al_2O_3 particles, 7 microns in diameter, are dispersed in the polycrystalline AgCl. The present investigation is designed to investigate whether larger cavities, produced mechanically, can similarly increase the fracture toughness of brittle materials. Since cavities also lower the weight of a structure, it is hoped that the toughness-to-weight ratio of a brittle material can be raised in this manner. These cavities have been introduced into notched and un-notched specimens of iron-3% silicon by drilling fine holes (diameter 200 microns or greater)prior to testing in tension, bending, and impact. The iron-3% silicon alloy was chosen because strain distributions around the cavities can be easily revealed by dislocation etch-pitting procedures. Furthermore, the holes can be drilled at room temperature where the alloy is ductile and tests can subsequently be carried out at low temperatures where the alloy is brittle. Cylindrical drilled holes have been chosen as the second phase because 1) they are easily introduced in controlled arrays and 2) the elastic stress field is simple and symmetrical about the hole's axis, so that theoretical calculations can be made with a minimum of difficulty.

While cavities increase a material's resistance to crack propagation, they also may decrease its resistance to crack nucleation. Local plastic strain, either twinning or slip, occurs around the cavities at stresses below the general yield stress because of the concentration of stress about the cavity. At temperatures below the ductile-brittle transition, these strains can cause cleavage crack nucleation. When the cavities are in the form of sharp notches, crack nucleation and fracture occur at stresses much below the general yield stress of the material ahead of the notch(5). If cylindrical holes also produce a drastic reduction in the stress required for cleavage crack nucleation, then the beneficial effects of holes on crack propagation and fracture toughness may be nullified. Consequently, this program will determine the effect of holes on crack nucleation as well as on propagation.

a) The effect of one hole and arrays of holes on the stress and strain required for cleavage crack nucleation.

Two heats of an iron-3% silicon alloy have been warm and cold rolled to a final sheet thickness of 0.040", roll-straightened, and sheared into tensile specimen blanks. A master template was designed and purchased so that tensile specimens may be rapidly machined on the laboratory's "Tensile-Kut" machine. Holes, whose diameter varied from 0.007" up to 0.060", were drilled into the guage length of the specimen (dimensions 1.25" x 0.400" x 0.040"), The specimens were annealed for various times at temperatures between 700 and 1200°C to produce structures whose ferrite grain size ranged from 5×10^{-4} to 5×10^{-2} inches diameter.

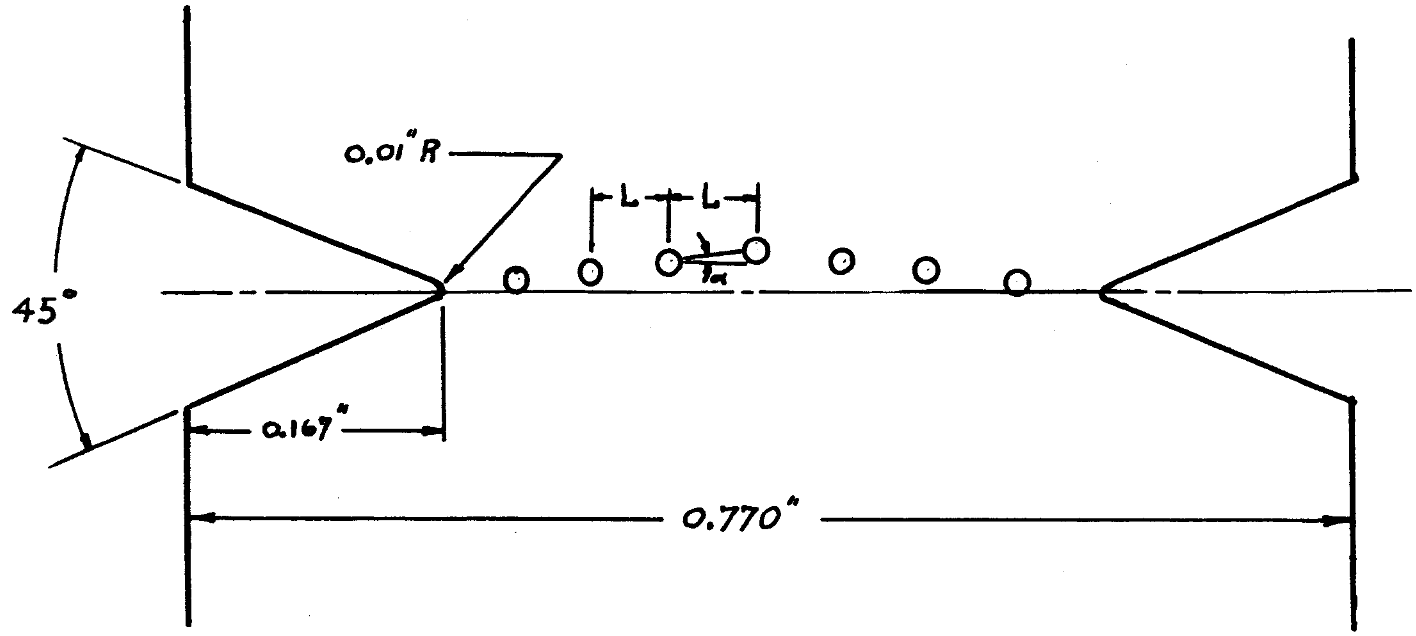


FIG. 1a

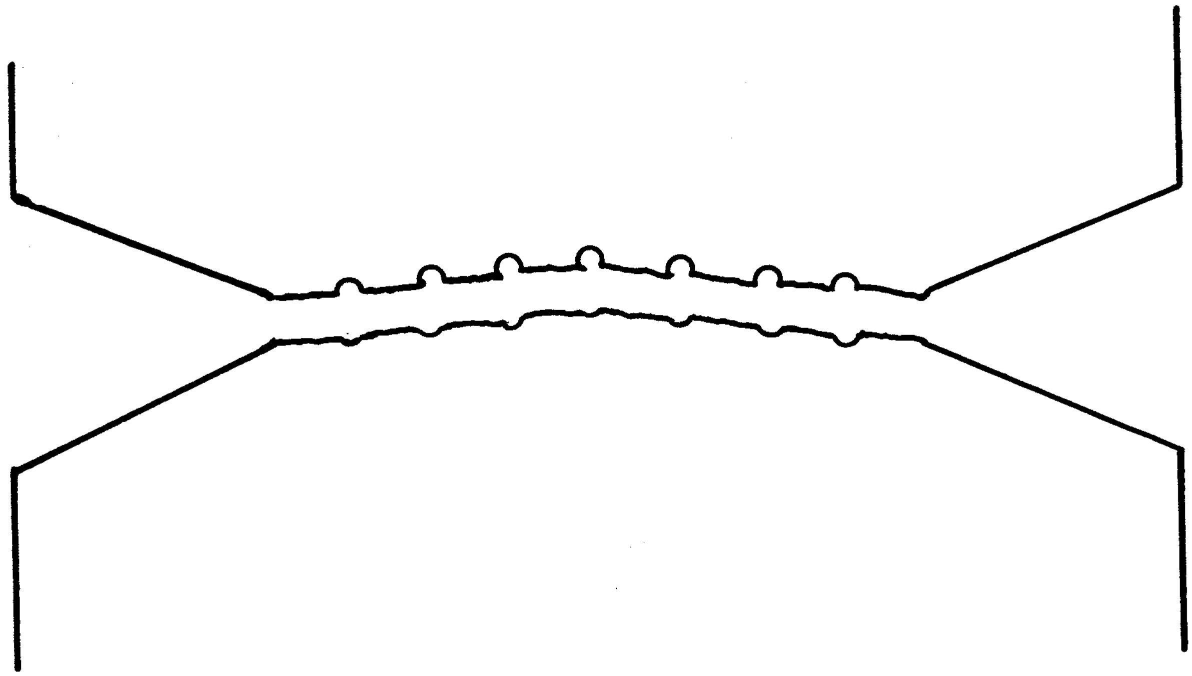


FIG. 1b

FIG. #1 - THE EFFECT OF HOLES ON FRACTURE PATH

Annealing treatments were performed in vacuum, in a furnace constructed for this program. Following annealing, the specimens were mechanically and electrolytically polished. They were then strained in tension at 77°K in a cryostat and gripping device built by Mr. Rau.

At the present time, we are studying the effect of hole size to grain size ratio on the ductility and fracture strength of iron-3% silicon at 77°K. Preliminary results indicate that when the hole size is of the order of the grain size, the hole has little effect on the net section fracture strength or ductility. When the ratio of hole size to grain size is increased, either by increasing the hole size or by refining the grain size, the hole causes a reduction in ductility. We do not have sufficient data to state whether the fracture stress is also lowered and this will be determined shortly. During the next month this part of the investigation will be completed. Similar studies will be carried out at a higher temperature where the alloy shows a ductile-brittle transition since cleavage is observed at all grain sizes at 77°K. Additional studies will be performed, using dislocation etch pitting techniques, of the size of the plastic zone in the vicinity of a hole, or arrays of holes, as a function of applied stress. This is necessary to achieve a quantitative understanding of the process of crack nucleation, which undoubtedly takes place inside the plastic zone[5].

b) <u>The effect of one hole and arrays of holes on the initiation and propagation of a crack at the root of a sharp notch</u>.

Symmetrical sharp notches (.167" deep, .010" root radius, 45° inducted angle), and arrays of holes were introduced into a sheet specimen as diagrammed in Figure 1a. The specimen was fractured at 77°K.

The fracture path followed the trace of the holes, as shown in Figure 1b. This preliminary result indicates that mechanically induced holes do have an effect on crack initiation and propagation at the root of a notch. In the following months we shall investigate whether:

a) the ductile-brittle transition temperature can be altered by the presence of arrays of holes such as those shown in Figure 1.

b) whether this alteration depends on the angle α between the holes,

c) whether the alteration depends on the size of the holes,

d) whether the fracture stress is influenced by the hole size and by α.

Crack initiation at the root of a notch depends on the attainment of a critical plastic displacement at the root, which in turn depends on the distance that yield zones spread from the root[6]. If holes interfere with the spreading of plastic zones, then they may increase the stress required for crack initiation at the root. This point will also be investigated using dislocation etch-pitting techniques.

3) <u>The Initiation of Fracture in a Composite Material Containing a Pre-Induced Crack</u> (with D. Barnett, graduate student).

Bilby, Cottrell, and Swinden[6], Dugdale[7], and others[8,9] have investigated the problem of fracture initiation at the tip of a pre-induced crack in a single phase material. They showed that as the applied stress T increases up to the general yield stress Y, yield zones spread a distance a - c from the tip of a crack of length 2c, where

$$\frac{c}{a} = \cos\left(\frac{\pi}{2}\frac{T}{Y}\right)$$

It was also shown that the crack will reinitiate and spread when a critical displacement ϕ^* (c) is produced at the tip, where

$$\phi^*(c) = \frac{4Yc}{\pi G} \ln\left({}^{a^*}/c\right)$$

G is the shear modulus and a* is the critical plastic zone size that must be produced.

The purpose of this theoretical investigation is to determine the criteria for the initiation of fracture in a two-phase material containing a crack of length 2c in one of the phases. It is assumed that both phases have the same elastic constants and lattice parameters and are isotropic. Suppose (Figure 2) that Y_1 and Y_2 are the yield stresses of phases (1) and (2), with $Y_2 > Y_1$. 2c is the crack length and 2ℓ is the width of phase (1). Phase 2 is infinitely wide. S_1 is the plastic zone size in phase (1), S_2 is the plastic zone size in phase (2), so that $a - c = S_1 + S_2$. T is the uniform tensile stress applied at infinity.

Using the Dugdale approach[7] of superposition, our calculations, which will be detailed in the next report, indicate that

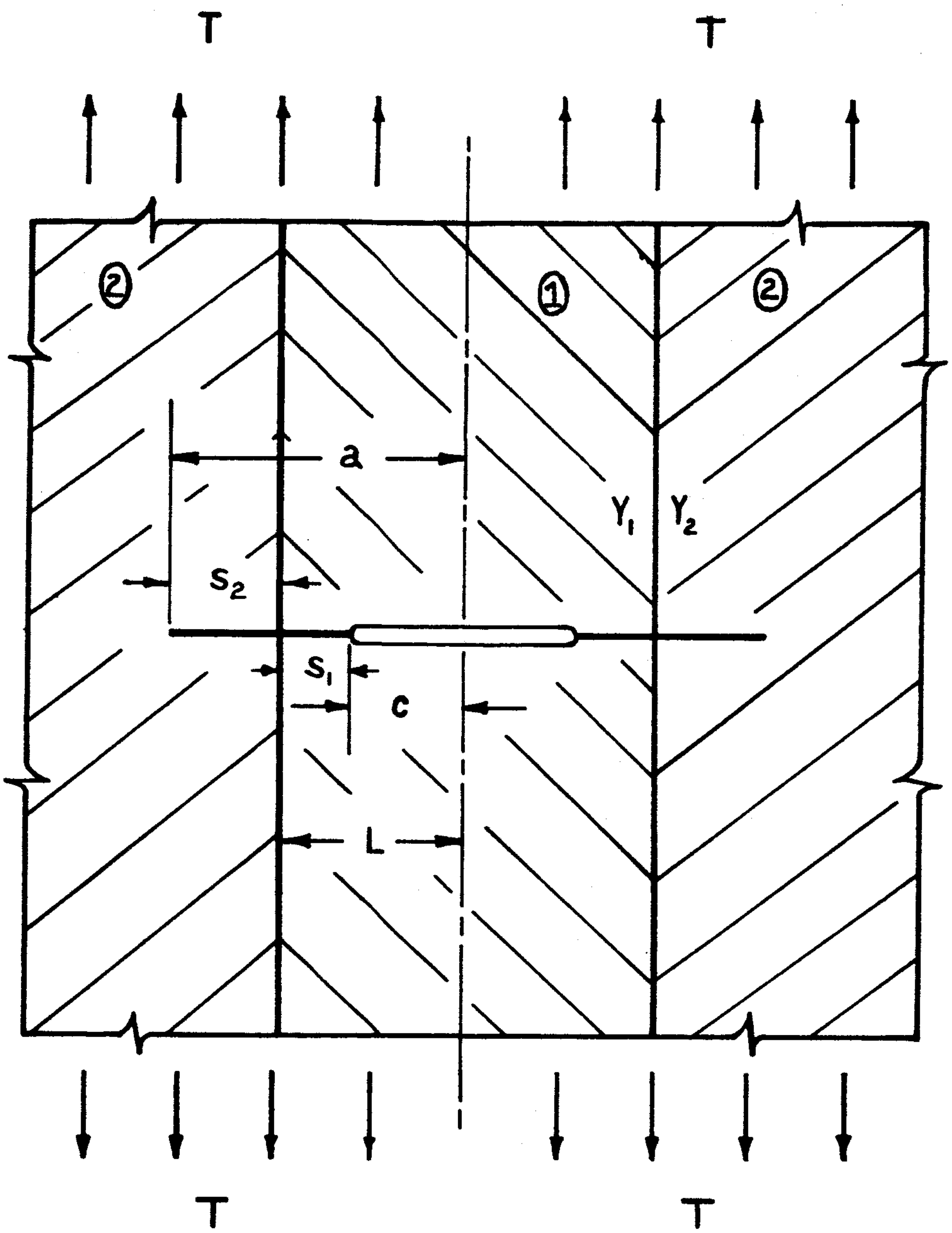
$$\theta_2 + \left(\frac{Y_2}{Y_1} - 1\right) \theta_3 = \frac{\pi T}{2Y_1}$$

where $\cos \theta_2 = c/a$

$\cos \theta_3 = \ell/a$

Physically, this equation implies that:

1) for $\frac{T}{Y_1}$ and c/ℓ held constant, ℓ/a increases as Y_2 increases, so that the plastic zone radius a decreases as the yield stress of



SYMMETRICAL DUGDALE CRACK AND PLASTIC ZONE IN A 2-PHASE INFINITE SOLID LOADED IN UNIFORM TENSION AT INFINITY

FIGURE 2

phase (2) increases.

2) For $\frac{T}{Y_1}$ and Y_2/Y_1 held constant, the plastic zone radius a increases as c increases.

3) If $\frac{\pi T}{2Y_1}$ is less than $\cos^{-1}(c/\ell)$, then a $\bullet$ S_1 and the plastic zone is entirely contained in phase (1).

Mr. Barnett holds a Ford Foundation Fellowship and has been working on this problem 1/8th time. During the following six months he will consider the displacements $\emptyset$ that are produced at the crack tip in the composite and the criteria for fracture at the crack tip. He shall also apply the Bilby-Cottrell-Swinden approach[6] for cracks loaded in anti-plane strain to determine the criteria for their re-initiation in the composite.

REFERENCES

1. J. Gurland, Trans AIME, 227, 1146 (1963).
2. See Figure 5, C. Nishimatsu and J. Gurland, Trans. ASM, 52, 469 (1960).
3. C. McMahon, Ph.D. Thesis, MIT, 1964.
4. T. L. Johnston, R. J. Stokes, C. H. Li, Trans. AIME, 221, 792 (1961)
5. J. Knott and A. Cottrell, J. Iron Steel Inst., 201, 249 (1963).
6. B. Bilby, A. Cottrell, K. Swinden, Proc. Roy. Soc., 272A, 304, (1963).
7. D. Dugdale, J. Mech. Phys. Solids, 8, 100 (1960).
8. A. Rosenfeld and G. Hahn, Acta Met., to be published.
9. J. Hult and F. McClintock, 9th Int. Conf. Appl. Mech., 8, 51 (1957).